OXFORD CRYOSYSTEMS

Cobra - Non Liquid Nitrogen Open Flow System

Operation & Instruction Guide

OXFORD CRYOSYSTEMS COBRA

Operation & Instruction Guide v3.1

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1 Introduction

Welcome to the Cobra operating and instruction guide. The Cobra is designed for use in the freezing of macromolecular, small molecule and powder samples during the collection of x-ray data. It can be used for all applications from shock cooling to lengthy data sets lasting many months.

Its versatility and flexibility means it can be fitted to practically any x-ray system including an Image Plate System, CCD Detector, Kappa Diffractometer or Powder Diffractometer. It is capable of cooling samples to 80 K in an open stream of dry nitrogen gas and heating them up to 400 K. Icing and unwanted moisture is prevented from reaching the sample by a concentric shroud of dry nitrogen or air at room temperature surrounding the inner core of cold gas.

1.1 Please register your Cobra system!

In order to help us provide technical support, we need you to register your Cobra system. You will need to fill in the **WARRANTY CERTIFICATE** attached at the end of this manual and faxing or emailing this back to us. This is very important as it allows us to track your enquiry and tie this up with the technical notes we have on your particular system.

1.2 How the Cobra works

The Cobra system consists of the Cobra refrigerator and gas delivery head, the controller unit and a helium compressor called a Cryodrive. The system runs on nitrogen gas and an optional nitrogen gas generator can be purchased as part of the Cobra system. Contact Oxford Cryosystems for more information. Figure 1 illustrates the gas flow circuit of a Cobra system.

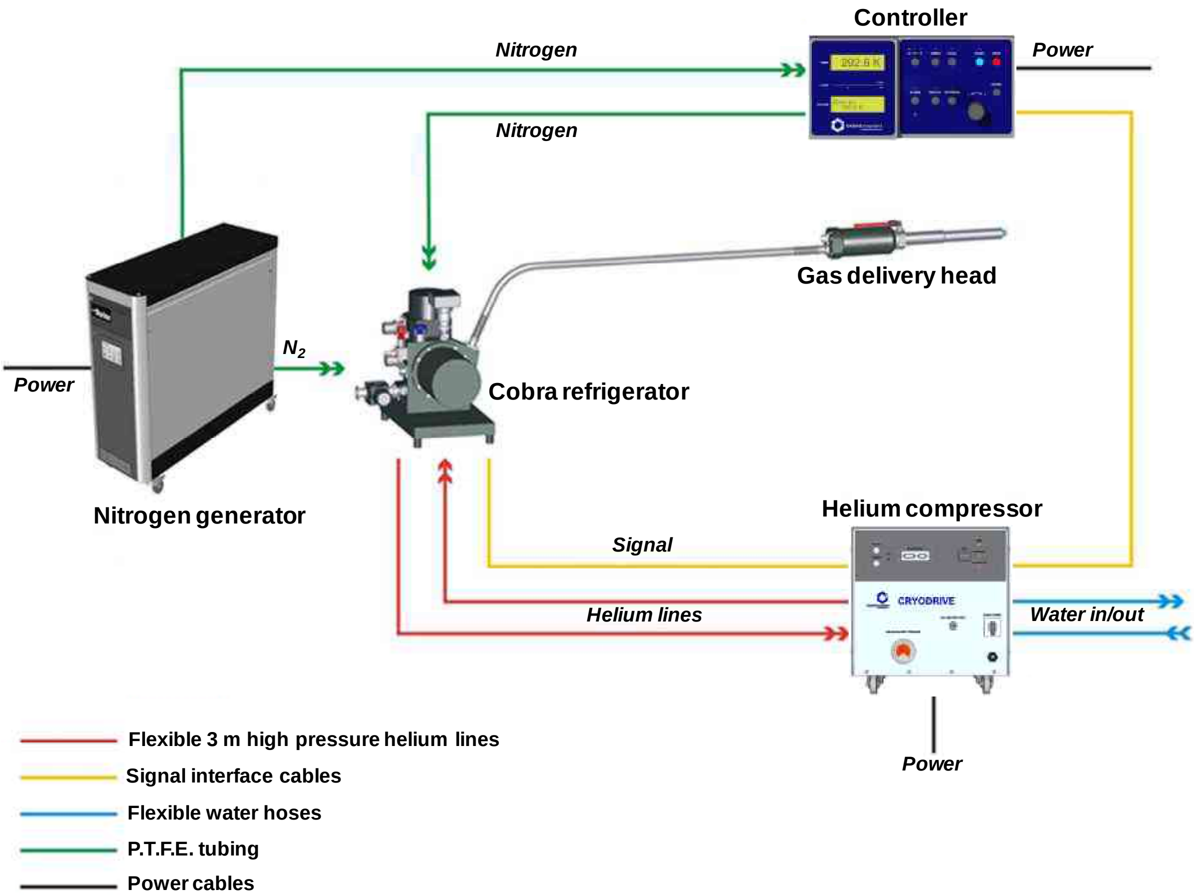


Figure 1 – Layout of Cobra gas flow circuit

Nitrogen gas is cooled by passing it through heat exchangers mounted on a closed cycle cooler. The cold nitrogen gas then passes out of the Cryostream style Cobra nozzle and over the sample.

The closed cycle cooler is mounted within the refrigerator section and operates using compressed helium gas provided by the Cryodrive compressor, which is water-cooled. The helium in this circuit is unrelated to the cold flow and is recycled by the compressor.

1.2.1 Cobra refrigerator

The Cobra refrigerator works using a Gifford McMahon Cryocooler (see Figure 2) and is used as part of a cryogenic cooling system with a helium compressor connected via interconnecting helium gas lines (see Section 1.2.2). For preventative maintenance of the Cobra refrigerator see Section 7.1.

Figure 2 – Cobra refrigerator with gas delivery head

1.2.2 Helium compressor

The Cryodrive supplies compressed helium gas in a closed circuit to the coldhead. High pressure helium gas provided by the Cryodrive is cyclically expanded by the coldhead to low pressure and returned to the compressor for recompression. The Cryodrive compressor also provides power for the coldhead motor via an interconnecting motor cable.

Figure 3 – Front and back of a Cryodrive helium compressor

The Coolstar coldheads are fitted with a variable speed stepper motor to drive the rotary valve and the speed of this valve is controlled via the Cryodrive, which in turn, is controlled by the Cobra controller and controls the cooling power of the coldhead (see Section 3).

The Cryodrive has one helium inlet (from the coldhead return), one helium outlet (to the coldhead supply) and sockets COLDHEAD 1 and COLDHEAD 2 for attachment of the Cobra refrigerator.

For more detailed information, a 'Cryodrive Operation and Instruction Guide' is also supplied with this Cobra manual although all information for set-up and running of a Cobra is described in this manual.

1.3 Sources of nitrogen gas

The Cobra can deliver the nitrogen gas at the desired temperature with a stability of 0.1 K while keeping the nitrogen gas consumption to a minimum. There are a variety of possible sources of nitrogen gas that can be used with the Cobra. All these possible gas sources must qualify to the following standard:

- Delivery pressure of 1 bar/14.5 psi (max 1.4 bar/20.3 psi)
- 12-15 L/minute gas flow for the inner cold stream
- Upwards of 15 L/min for the outer warm stream (if dry nitrogen is to be used)
- Dew point of better than -65°C
- Nitrogen purity of at least 95%

Generally, three sources of nitrogen gas can be considered to supply nitrogen to the Cobra:

1. An in-house source usually supplied from the boil off from a storage tank at your facility. It is prudent to check on the reliability of this flow and to make sure that is has been plumbed into the laboratory through clean and dry piping.

2. A nitrogen gas generator can be used to supply the gas. These generators are readily available and can also be purchased through Oxford Cryosystems. The units usually consist of an oil free air compressor feeding into a membrane filtration system or a pressure swing adsorption (PSA) system which separates out the nitrogen from the oxygen, CO_2 and water vapour.

3. Liquid nitrogen boil-off from a liquid cylinder. These are basically liquid nitrogen storage vessels and boil-off the liquid at a high enough rate to produce a reliable gas flow. A 350 litre tank would last up to 22 days if an AD51 Dry Air Unit was used for the outer stream and up to 11 days if this source were also used to supply the gas for the outer stream.

1.4 Items required for assembling your Cobra

NOTE

The Cobra system is normally assembled and set-up by an Oxford Cryosystems engineer or an engineer previously trained by Oxford Cryosystems. Before attempting any installation yourself please contact Oxford Cryosystems for more information.

The component parts of the system are:

- ✓ The integrated Cobra gas delivery head and refrigerator unit
- ✓ 700 Series Cobra controller
- ✓ Interconnecting tube-set including:
    - One 10 m stainless steel 6 mm flexible tube (nitrogen source to controller)
    - One 2 m stainless steel 6 mm flexible tube (controller to refrigerator)
    - One 10 m 6 mm PTFE tubing (nitrogen source to outer shroud flow meter)
    - One 1 m 6 mm tube (shield stream delivery)
- ✓ Cryodrive cable
- ✓ Black cable junction box (attached to refrigerator base)
- ✓ Cryodrive compressor
- ✓ Stainless steel flexible helium lines (x2)
- ✓ Manual pack
- ✓ Outer stream flow kit containing 2-10 L/min flow meter with Varibeam fixing bracket and one length of 5 m 8 mm tubing
- ✓ One serial cable for Cryopad
- ✓ One Cryopad CD

Optional extras include:

- ✓ Nitrogen generator
- ✓ Cobra assembly stand
- ✓ Varibeam gas delivery head support stand

2 Setting up

2.1 Mounting of the Cobra gas delivery head on the x-ray system

The mounting of the Cobra gas delivery head depends on the particular x-ray system being used.

NOTE

The following rules and instructions are guidelines only and if a user has an alternative technique for mounting the Cobra then they are welcome to use it. If the user is in anyway unsure of the mounting of the Cobra they should contact their local Cobra supplier or Oxford Cryosystems for advice.

2.1.1 General rules

There are a few general rules the user should consider when mounting the gas delivery head and fixing the support stand in position.

1. Do not point the cold stream directly at the detector.

2. Try to limit the amount the Cobra nozzle infringes the path of the x-rays.

3. Do not point the cold stream at any optical device or gearing (these devices need to be more than 15 cm away).

4. Do not mount the cold stream coaxial with the goniometer head as this may cause icing problems.

5. Do not fix the stand to your cabinet top so it prevents access to your x-ray tube, prevents the detector being swung in theta or makes access to the crystal difficult.

6. If the nozzle of the Cobra is horizontal in the Varibeam and the system is being run at 80 K, then incline the nozzle by a few degrees or ramp the system to 82 K. This will avoid liquid formation in the nozzle due to temperatures in the refrigerator being close to 77.4 K.

2.2 Order of assembly

2.2.1 Matching the Cobra and 700 Series Cobra controller

The Cobra refrigerator and 700 Series controller are supplied as a matched pair. The individual characteristics of the gas delivery head sensors are programmed into the controller. Please contact Oxford Cryosystems if you believe you may have unmatched units.

2.2.2 Operating voltage

It is essential that the Cobra is configured to operate on the local mains electrical supply.

Item	Operating voltage requirements
Cobra controller	The voltage selector switch on the rear panel must be set to the correct position, 200-240 Volts AC, 50 Hz, 3 Amps 100-120 Volts AC, 50-60 Hz, 6 Amps. Make sure an 'Anti-Surge' (T) type fuse of the correct rating is fitted.
Cryodrive	User configurable supply voltage: 200, 220 or 240 V at 50 Hz or 200, 208 or 220 V at 60 Hz Supply voltage tolerance: +10 % Maximum supply fuse rating: 30 A Recommended CryoController fuse type: Slow blow Maximum CryoController fuse rating: 5 A Over voltage category (IEC664): 2

Oxford Cryosystems ships all Cryodrives set to 240 V and 50 Hz as this is how they are commissioned in the UK. It will be necessary to refer to Section 2.2.3 to set the Primary Tap Connections for the local voltage and frequency.

2.2.3 Electrical supply connection for Cryodrive

We recommend that you use a suitably fused isolator at your electrical supply outlet. Locate the isolator switch close to the electrical outlet. It is also recommended that back-up fuses are installed at electrical supply outlet. You must configure the Cryodrive to suit your electrical supply. The Cryodrive is dispatched configured for use with 240 V and 50 Hz electrical supply.

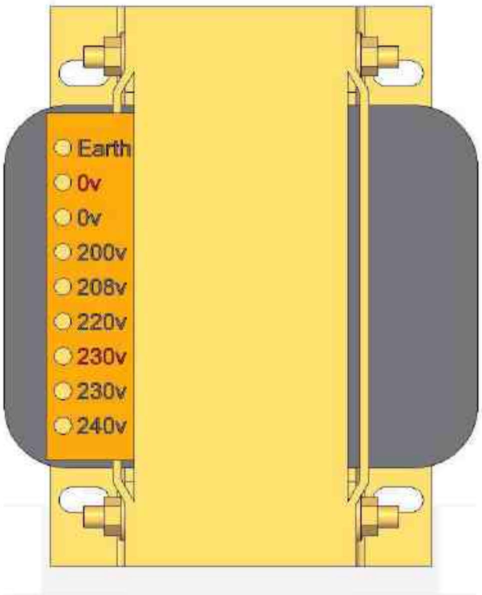


Figure 4 – Transformer showing different electrical supply connections

Use the procedure below to change this configuration.

- Look at Table 1: find your electrical supply in the left-hand column, look along this row to find the connections you must make for the wire 30(W30) and wire 31(W31).

- Remove the lid of the Cryodrive and locate the transformer.

- With reference to Figure 4, change the position of wire 30 (W30) and wire 31 (W31) so that they are in the correct position for your electrical supply. Ensure the connections are fully tightened. **Do not move the wires attached to the red 0 V and 230 V connectors, as these provide power to the secondary stepper motor transformer and are not part of the procedure.**

- Locate the protection switch on the current-limit potentiometer which is situated in the CryoController, refer to Figure 5. The switch can be accessed by removing the lid of the CryoController unit.

- Look at Table 2: find your Cryodrive type and electrical supply frequency in the left-hand column, look along this row to find the recommended limit for the protection switch current.

- Use a small screwdriver to adjust the current-limit potentiometer to the recommended value.

- Replace the lid of CryoController and the Cryodrive.

- Make sure that the Cryodrive ON/OFF switch is in the OFF position and connect the Cryodrive to your electrical supply.

Electrical Supply	**Primary Tap Connection**					
	N	200 V	208 V	220 V	230 V	240 V
50 Hz, 200 V	W32	W31	W30	-	-	-
50 Hz, 220 V	W32	-	W30	W31	-	-
50 Hz, 240 V	W32	-	W30	-	-	W31
60 Hz, 200 V	W32	W31	-	-	W30	-
60 Hz, 208 V	W32	-	W31	-	W30	-
60 Hz, 220 V	W32	-	-	W31	W30	-
60 Hz, 240 V	W32				W30	W31

Table 1 – Wire connections to primary tap connections

Cryodrive model	Electrical supply voltage			
	200V	208V	220V	240V
Cryodrive 1.5, 50 Hz supply	11A	-	10A	10A
Cryodrive 1.5, 60 Hz supply	11A	11A	11A	-
Cryodrive 2.0, 50 Hz supply	14A	-	13A	12A
Cryodrive 2.0, 60 Hz supply	15A	14A	14A	-
Cryodrive 3.0, 50 Hz supply	16A	-	16A	16A
Cryodrive 3.0, 60 Hz supply	18A	17A	16A	-

Table 2 – Recommended protection switch current limit setting

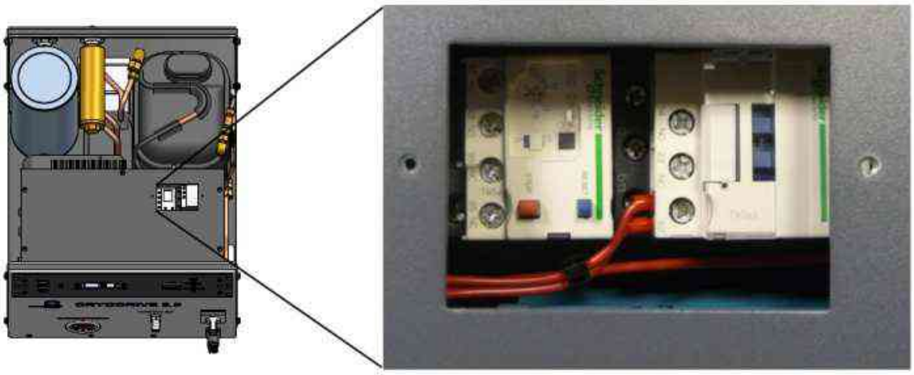


Figure 5 – Overload relay showing stop and reset buttons

2.2.4 Helium gas hose connection

WARNING

If you are unsure on how to connect the helium gas hoses, please contact Oxford Cryosystems. Failure to connect the hoses properly might result in complete loss of helium pressure within the Cryodrive.

The main layout is illustrated schematically on the diagram in Figure 1 and connections are generally labelled equivalently at each end. Start with the two high-pressure hoses between the Cobra refrigerator and the Cryodrive compressor module. The ends are colour-coded to avoid confusion: the helium return line has a GREEN band; helium supply line has a RED band, see Figure 6.

Figure 6 – Helium gas hoses

The Cryodrive has one helium supply outlet and one helium return inlet. The recommended procedures for connecting and disconnecting the couplings are described below (for more information read the Cryodrive Operation & Instruction Guide).

1. To prevent damage to the couplings and leakage of the helium, you must use two spanners as shown in Figure 7.
2. Complete the fitting as quickly as possible to prevent leakage of the helium from the couplings.
3. Note that helium hoses are pressurized with helium. When you fit these components, you must follow the safety advice and instructions given in the instruction manual supplied with them. Do not over bend or twist the helium hoses; do not allow damage to occur to the braid on the outside of the hoses.
4. Connect the hose marked with the red band to the helium supply connector on the Cryodrive. Connect the other end of the hose to the helium supply connector (marked with a red band) on the refrigerator.
5. Connect the hose marked with the green band to the helium return connector on the Cryodrive. Connect the other end of the hose to the helium return connector (marked with a green band) on the refrigerator.
6. Check that the connecting surfaces of the couplings are clean.
7. Check that the sealing ‘O’ ring is in place.
8. Connect the coupling halves by hand until you feel resistance.
9. Refer to Figure 7 and rotate spanner (green arrow) whilst holding the other spanner still (red arrow) to fully tighten the coupling.

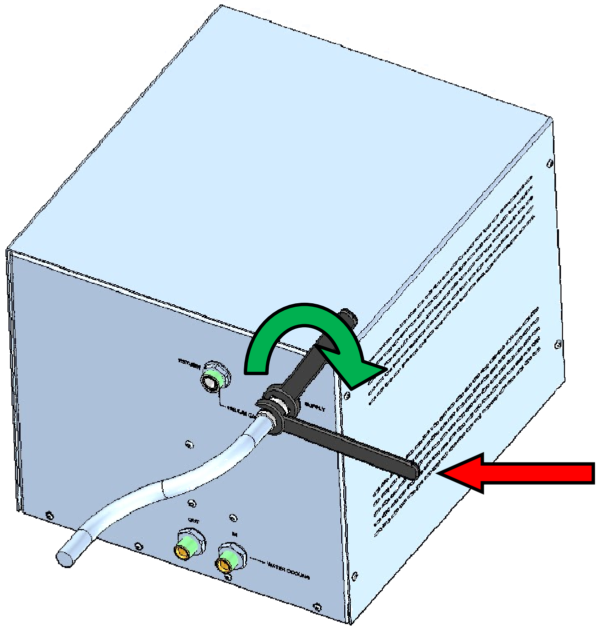


Figure 7 – Connection of a helium hose to the Cryodrive using two spanners

NOTE

When the connection is fully made, rotate one spanner in the opposite direction for one quarter turn to ensure that the sealing 'O' ring is not over compressed. If you over compress the sealing 'O' ring the service life of the fittings will be reduced.

2.2.5 Helium gas pressure check

The location of the pressure gauge is illustrated in Figure 8. Whilst the Cryodrive is running, the pressure indicated is significantly higher than the quiescent value of approximately 16.5 bar. Under normal operating conditions it should be about 22 bar and will noticeably oscillate by around 0.5 bar. It is good practice to monitor the Cryodrive charge pressure, especially if the system has been dismantled and reconnected recently, as gas can be lost during the attachment and disconnection of the high-pressure hoses. As mentioned before, if the pressure is observed to have fallen, contact Oxford Cryosystems to arrange a re-charge.

IMPORTANT – If the Cryodrive is used for the first time

It is important to check the charge pressure of the Cryodrive compressor. In the quiescent state, the pressure should read approximately 16.5 bar ±1.0 bar (240 psi ±15 psi). If this pressure reads less than 15.5 bar (220 psi), contact Oxford Cryosystems. This check should be repeated, if possible, each time unit is to be turned on.

Figure 8 – Front of Cryodrive showing the pressure gauge

2.2.6 Cooling water connection

1. Use hose clips to secure suitable water hoses (1/2-inch nominal internal diameter) to the water connection nozzles.
2. Connect the supply and return hoses to the cooling water inlet and outlet connectors as marked on the rear of the Cryodrive.
3. Connect the water supply hose to cooling water supply with an adequate flow rate and temperature (see Section 2.2.7).
4. Connect the water return hose to a suitable drain.
5. Turn on the cooling water supply and check that there are no water leaks.

2.2.7 Water cooling requirement

- Water cooling/chiller requirement: 3.0 kW
- Minimum flow rate: 1.5 L/min
- Maximum flow rate: 7.0 L/min
- Maximum water supply pressure: 101.5 psig.
- Minimum water supply temperature (at start-up): +8ºC.
- Maximum water discharge temperature: +33ºC.
- Water quality pH range: 6.0 to 8.0.
- Maximum calcium carbonate concentration: 75 ppm.

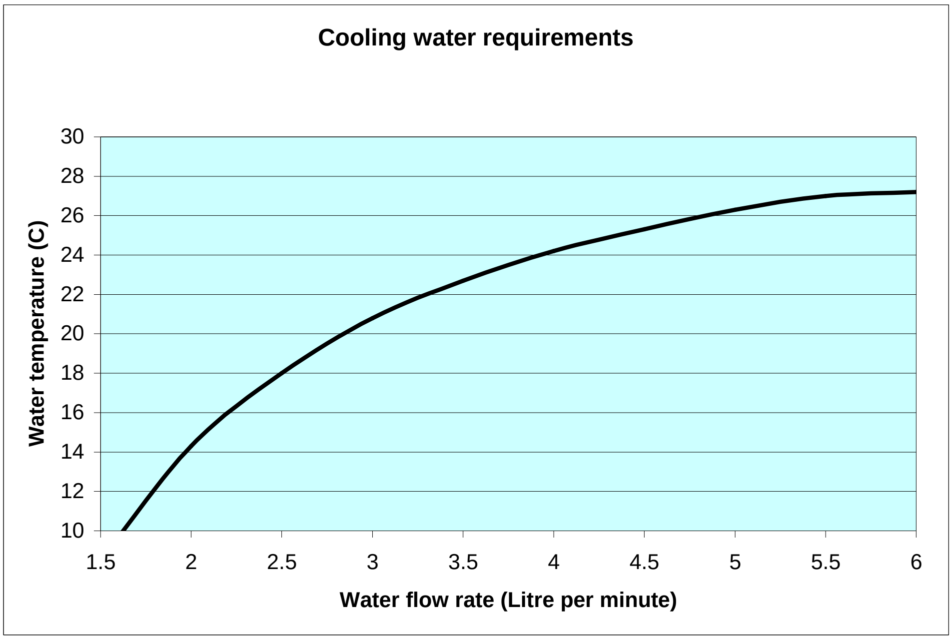


Figure 9 – Cooling water requirements for the Cryodrive

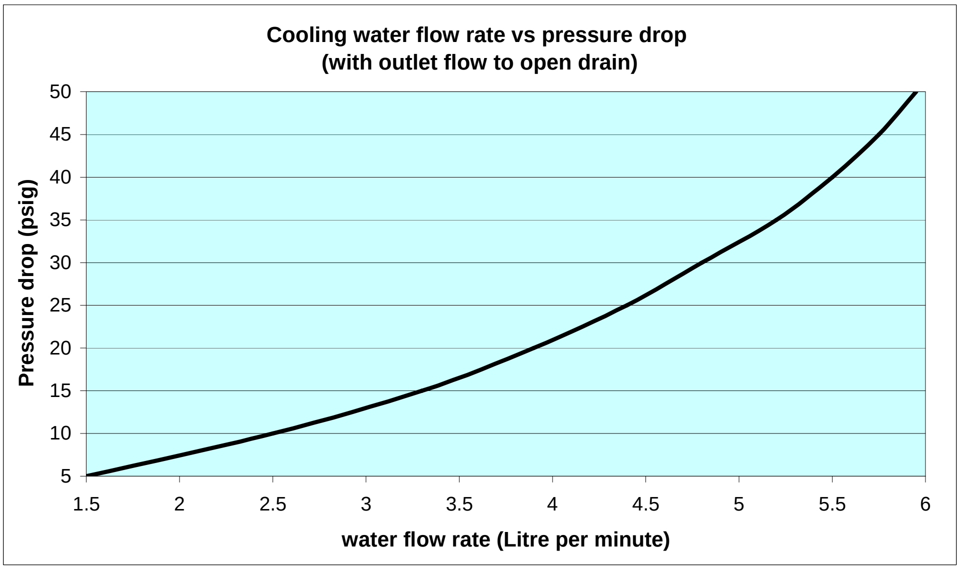


Figure 10 – Cooling water flow rate vs pressure drop

2.2.8 Nitrogen gas connections

Next fit the gas delivery tubes. The nitrogen exchange gas must be dry and set to 1 bar gauge output. Use the following convention for fitting each tube into its connector.

Tube type	Connector /location		Connector /location
10 m 6 mm stainless steel tube	Nitrogen supply (left port on the back of the N_2 generator, if included)	connected to	IN on line drier
0.5 m PTFE tube with quick fit connectors	OUT on line drier	connected to	NITROGEN IN on controller
2 m 6 mm stainless steel tube	FLOW OUT on controller	connected to	Nitrogen input on refrigerator
10 m 6 mm PTFE tube	Nitrogen supply or dry air unit	connected to	Bottom 6 mm fitting of outer stream flow kit flow meter
Short 6 mm red tube	Top 6 mm fitting of outer stream flow kit flow meter	connected to	Nozzle outer stream connector

Connect the signal cables and the mains power cables. The COLDHEAD 1 and PCSP sockets are at the front of the Cryodrive enclosure.

2.2.9 Use of an Oxford Cryosystems line drier unit with the Cobra

When using a Cobra for extended periods of operation (>one month) it is necessary to ensure that the nitrogen gas being used with the system is free from potential impurities that may give rise to blockages in the Cobra gas delivery head over time.

Although most nitrogen sources are extremely pure and clean, the Cobra system can filter more than 250,000 Litres of gas per month and any tiny traces of either water vapour or carbon dioxide can accumulate in the heat exchanger and block the unit. These impurities arise from the manufacture and transport of the nitrogen to the Cobra and, even though the Cobra gas circuit is positively pressurised preventing inward leaks, impurities can still affect the system. The blocks form in the FLOW OUT path of Cobra gas delivery head as the nitrogen gas is cooled from room temperature to liquid nitrogen temperature in the heat exchanger.

To ensure that the Cobra performs as required, some basic rules should be followed:

- The nitrogen gas should have an atmospheric dew point of lower than -65°C. Levels as low as this are often difficult to measure without the correct equipment. If you are unsure, contact Oxford Cryosystems.
- The nitrogen gas should be carried to the Cobra through clean, dry and non-permeable piping or tubing. Good examples are stainless steel or copper. Plastic tubing is not really suitable, although PTFE is the only viable candidate. **Do not use nylon tubing**.

2.2.9.1 BEFORE FITTING AND USING YOUR LINE DRIER

When the Cobra is first installed run your system without fitting your line drier. If there are any impurities in the nitrogen, the Cobra will block over a period of time. When first commissioning the Cobra, this time period should be checked to ensure the quality of the nitrogen. To provide some loose guidelines, an in-house nitrogen gas generator should run for five weeks without a line drier. Other in-house sources may be as low as three weeks. This is acceptable.

Once the system has run for a satisfactory period without shutting down, fit the line drier unit.

2.2.9.2 FITTING AND USING THE LINE DRIER

The line drier is designed to remove traces of water vapour and carbon dioxide from the nitrogen stream before a block can form. **It will not cope with gross contamination of the nitrogen supply.**

Check that all the quick-release connectors are clicked into place correctly.

Remove the blanking plugs, (these plugs must be fitted to the line drier during transit and storage). Fit the line drier unit behind on the controller on the GAS IN line. This is between the controller and the nitrogen gas source. **Do not leave the line drier open to the atmosphere to avoid contamination.**

For further instructions and information on the line drier, see the Tech Note supplied with each line drier unit.

2.2.10 How to switch the Cobra Controller on

The Cobra Controller is designed to provide a completely flexible means of controlling temperature. This is achieved by allowing the user to enter multiple phases.

Ensure the Cryodrive compressor has adequate cooling water (consult the *Cooling Water Connection* section on the previous page or the Cryodrive manual for exact cooling water requirements). Press the 'ON' switch for the Cobra Controller, which is on the right hand side of the rear panel of the controller.

Now switch the Cryodrive on using the large switch on the front right hand side of the Cryodrive. If the unit has been connected up correctly, the Cryodrive should initialise, but not start running. This is because the action of being plugged into the Cobra Controller holds the Cryodrive off.

2.2.11 Cobra initialisation and screen options

As the controller is switched on, it undergoes an initialisation process and a self-check procedure. During the self-check, the controller checks to make sure all parts of the Cobra system are connected and working properly.

Figure 11 – Front of a Controller

The various buttons that can be seen on the controller (Figure 11) are described below.

- TEMP screen. Whilst the Cobra is running this screen displays the temperature of the gas stream. During start-up and shutdown the screen is used for status messages.

- FLOW meter. The flow meter indicates the nitrogen gas flow in L/min. If no lamps are illuminated, the gas flow is zero. The first lamp indicates 5 L/min, all six lamps indicates 10 L/min.

- READY lamp. This lamp is designed to report the status inside the refrigerator. When the system is going through its cool down process, the Cobra reduces the flow to 1 L/min to take the heat load off the refrigerator so it can get cold as quickly as possible. In this situation, the READY lamp is off. Once the inside of the refrigerator is cold enough, the READY lamp will illuminate and the flow will automatically increase to its normal operating value.

- STATUS screen. This screen displays information described in detail under the heading *Display Modes*.

- K/ºC/ºF button and lamps. The button allows the temperature units used by the Cobra to be switched between Kelvin, Centigrade and Fahrenheit **at any time**. The current choice of units indicated by the illuminated K/ºC/ºF lamp. For the purposes of this manual, temperatures are indicated in Kelvin (K).

- TURBO button and lamp. The button allows the nitrogen gas flow rate to be adjusted between a normal value (5 L/min except at temperatures below 90 K) and a 'Turbo' value (10 L/min except above 310 K). If the TURBO lamp is OFF this indicates normal flow, whereas ON indicates Turbo flow.

- HOLD button and lamp. Pressing the HOLD button will execute a Hold (see Section 3) and illuminate the HOLD lamp. If Cobra is already in a Hold, pressing HOLD again will release it.

- START button and lamp. The START button switches the Cobra on, executing the start-up phase or the current Phase Table (see Section 3). This button is also used to re-start the control program after it has been halted.

- STOP button. The STOP button will immediately halt the Cobra, turning off the Cryodrive and all the heaters. The controller may then be safely switched off, or else re-started by pressing START.

NOTE

The approved method of shut down is via an END phase

- ALARM button and lamp. If an alarm condition develops (see section 3.2.4 *Alarm Conditions*), the ALARM will be illuminated and a buzzer may sound. Pressing the ALARM button will display the cause of the alarm in the STATUS screen, and will also cancel the buzzer.

- DISPLAY button and lamp. The DISPLAY button is used to toggle the Display Mode (see *Display Modes*), indicated by the corresponding lamp.

- PROGRAM button and lamp. The PROGRAM button is used to toggle Program Mode (see Section 3), indicated by the corresponding lamp.

- IntelliKnob. This knob is used to scroll the contents of the STATUS screen. In Program Mode it is also used together with the ENTER button to input information. The IntelliKnob is speed sensitive. This means the faster the Knob is turned, the greater the increment in the number and the slower the Knob is turned, the smaller the increment in the number.

- ENTER button. This button is used during Program Mode (see *Programming the Cobra*) to input information.

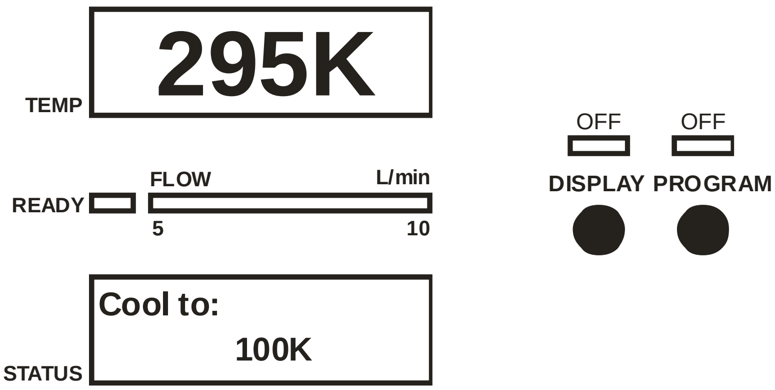
2.2.12 Display modes

The STATUS screen displays a variety of information depending on the Display Mode and whether Cobra is **Running** or **Idle**. In each case the contents of the STATUS screen may be scrolled using the IntelliKnob.

The various situations are summarised below.

2.2.12.1 DISPLAY MODE 1

Cobra State:
IDLE (Power on, not running)
DISPLAY Lamp: **OFF**
PROGRAM Lamp: **OFF**

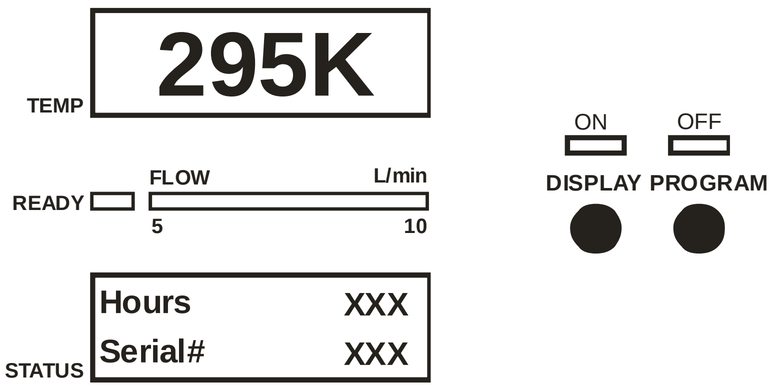


Description:
<u>Idle phase table mode</u>
If a program has not been entered, use the IntelliKnob to adjust the temperature and press START to begin. The Cobra will then enter a COOL function and achieve the required temperature as quickly as possible. If a program has been entered, use the IntelliKnob to scroll the STATUS screen and press START to begin.

Press DISPLAY or PROGRAM to enter the modes below.

2.2.12.2 DISPLAY MODE 2

Cobra State: **IDLE**
DISPLAY Lamp: **ON**
PROGRAM Lamp: **OFF**



Description:

Idle phase table
Use the IntelliKnob to scroll through the following information:

Vacuum	**mbar** vacuum inside refrigerator
Hours	The cumulative time the system has run since manufacture.
Serial #	Controller serial number
Software	The version of the controller software
Shutdown	This indicates the last reason for shutdown. Options are:

STOP	The STOP button has been pressed.
END	The system has been shut down due to a programmed END.
PURGE	The system has been shut down due to a programmed PURGE. (see *Further Programming on the Cobra Controller*)
POWER	The power has been switched off at the mains.
REFR T	If the Refrigerator temperature has failed to reach 100 K within an hour.
FLOW	Shutdown due to low flow.
T CTRL	There has been a large temperature error.
SENSOR	This indicates a sensor failure.
SINK	Controller overheating.
PSU	Power supply overheating.
LAST ERR	This stores the last reason for the shutdown but does not include STOP or POWER.

If there has been an unexpected shutdown, the following list of items is recorded on this list after the LAST ERR:

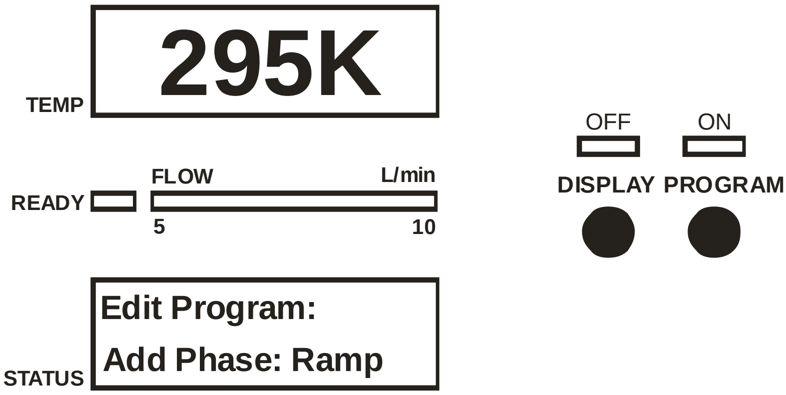
Set T, Gas T, Gas Heat, Refr T, Refr Heat, Vacuum, Cryo Speed, Cryo, Cryo Shift, Gas Type, Gas Flow, Pressure, Run Time.

These are recorded to allow the user to diagnose the reason for the shutdown and are stored until there is another erroneous shutdown.

Press DISPLAY or PROGRAM to alter the Display Mode.

2.2.12.3 DISPLAY MODE 3

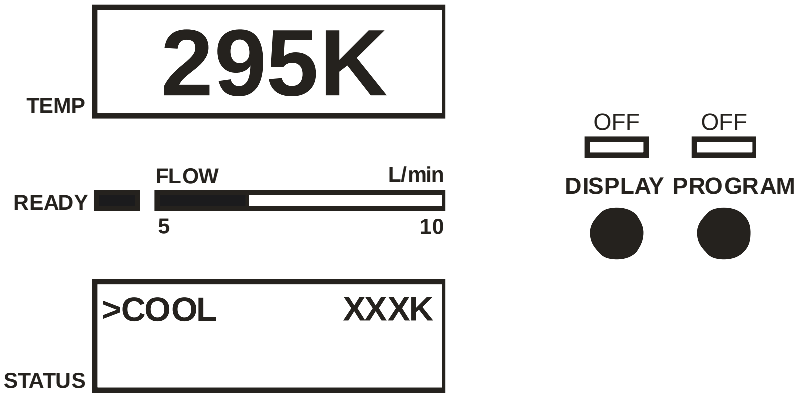
Cobra State: **IDLE**
DISPLAY Lamp: **OFF**
PROGRAM Lamp: **ON**



Description:
Program mode
This mode allows the user to program the Cobra as described in *Programming the Cobra*. The list of phases also gives you the option to save or load a program. 'Save Program' will save the current program and 'Load Program' will load the last saved program. Only one program can be saved.
Press DISPLAY or PROGRAM to alter the Display Mode.

2.2.12.4 DISPLAY MODE 4

Cobra State: **RUNNING**
DISPLAY Lamp: **OFF**
PROGRAM Lamp: **OFF**

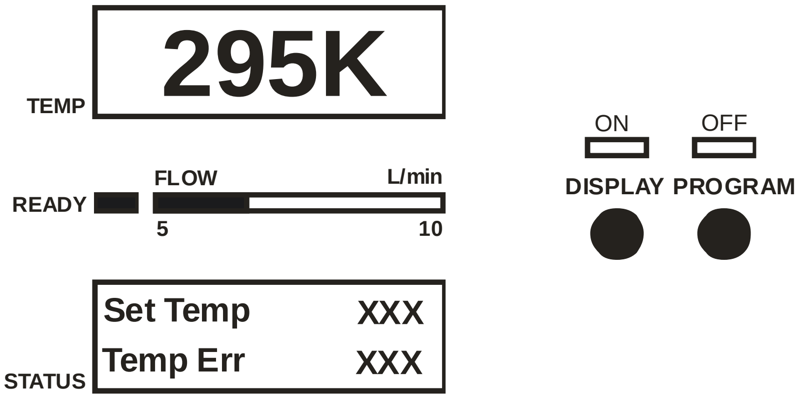


Description:
<u>Phase table mode</u>
The Cobra lists the phases in the current program, with the current phase at the top of the list. The current phase is indicated with a '>'. Use the IntelliKnob to scroll through the list.

Press DISPLAY or PROGRAM to enter the Display Modes 5 & 6.

2.2.12.5 DISPLAY MODE 5

Cobra State: **RUNNING**
DISPLAY Lamp: **ON**
PROGRAM Lamp: **OFF**

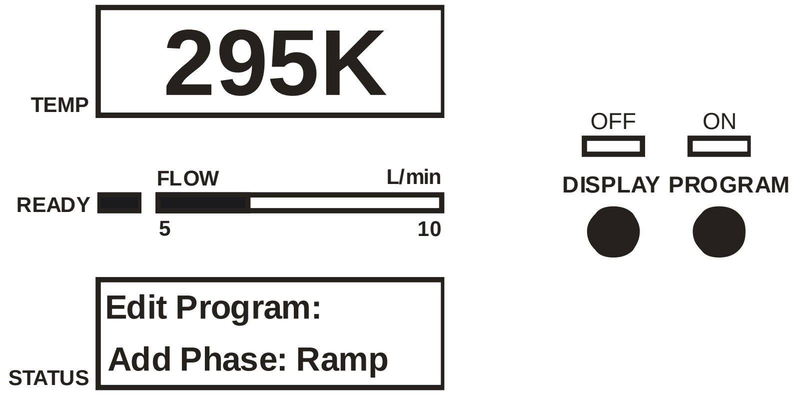


Description:
<u>Running display mode</u>

Set Temp	The temperature to which Cobra is controlling.
Temp Error	The difference between the Sample Temp and the Set Temp, except in a Cool phase, in which Temp Error is zero.
Run Time	How long system has been running since START was last pressed.
Flow Rate	Gas flow in L/min
Pressure	Back pressure in bars.
Gas Heat	The power to the sample heater, expressed as a percentage of full power. The instantaneous value is given and the average is in brackets.
Refr Temp	The temperature measured at the Cobra refrigerator.
Refr Heat	The power to the refrigerator heater, expressed as a percentage of full power.
Vacuum	The pressure measured in mbar by the vacuum gauge
Cryoshutter	
Cryo	Cryodrive status, (e.g. Running)
Cryo Speed	Cryodrive speed in RPM.
Cryo Shift	The amount by which the Cryodrive speed has been increased from its normal value in order to achieve rapid cooling. Press DISPLAY or PROGRAM to alter the Display Mode.

2.2.12.6 DISPLAY MODE 6

Cobra State: **RUNNING**
DISPLAY Lamp: **OFF**
PROGRAM Lamp: **ON**



Description:
<u>Program mode</u>
This mode allows the user to program the Cobra as described in *Programming the Cobra*. New phases are added at the end of the list of phases. If the system is in a HOLD, press HOLD to begin the next phase. Press DISPLAY or PROGRAM to alter the Display Mode.

3 Programming the Cobra

Switch the Cobra controller on according to the instructions above and wait for the system to initialise.

3.1 Using the quick start facility and the COOL function

To cool as quickly as possible simply rotate the IntelliKnob to the appropriate temperature and press START. The Cobra will remember the last value requested here and store it for the next time the Quick Start facility is used.

NOTE

The system uses the COOL function to get cold as quickly as possible. The COOL function runs the Cryodrive at high speed to cool the gas delivery head as fast as possible and reduces the flow to 1 L/min. Once the refrigerator is cold, the flow is increased to the desired level. **It is only possible to COOL down. It is not possible to COOL up in temperature. To raise the temperature of the gas, program a RAMP.**

Once the Cobra reaches the desired temperature, the controller will automatically enter a HOLD in the phase table.

With the system now running, press DISPLAY to view *Display Mode 5* discussed above. This displays all the parameters of the system. Alternatively, press PROGRAM to enter *Display Mode 6* and add additional phases to your program (see *List of Phases* and *Further Programming of the Cobra Controller*).

3.2 Further programming of the Cobra controller

While the system is idle or running, it is possible to program more detailed phases.

Press PROGRAM (this illuminates the PROGRAM lamp). The STATUS screen then displays the following:

```
Edit Program:
Add Phase: Ramp
```

Spin the IntelliKnob to see the available phases. Press ENTER at any time to accept a particular phase.

Here is a list of the possible phases and other parameters that each one requires.

Phase and Description	STATUS Screen Modes
Phase: RAMP <u>Description</u> Change temperature at a controlled rate. When ramping down in temperature, if the selected rate is too fast for the Cobra to follow, the Controller will automatically enter the RAMP/WAIT mode (this will be indicated on the screen). The effect of this is to stop the ramp in order for the gas temperature to catch up to within 5 K of the set temperature. The Ramp Rate may be anything between 1 and 360 K/hr.	**Edit Program:** **Add Phase: Ramp** **Ramp Rate:** **120K/hr** **Final Temp:** **100K**
Phase: COOL <u>Description</u> COOL is designed to get the system as cold as quickly as possible. It is not possible to spin the IntelliKnob above the end temperature of the previous phase or the current gas temperature. Note: It is only possible to COOL down. It is not possible to COOL up in temperature. To raise the temperature of the gas, program a RAMP.	**Edit Program:** **Add Phase: COOL** **Cool to:** **100K**
Phase: PLAT <u>Description</u> Maintain temperature fixed for a certain time. The user is prompted to enter a temperature at which to plateau and to specify the plateau's duration. Below 10hr 00min the PLAT function will start to count down in seconds and this will be displayed in the STATUS screen during running.	**Edit Program:** **Add Phase: PLAT** **Plat Length:** **1:00 (hh:mm)**
Phase: HOLD <u>Description</u> Maintain temperature fixed indefinitely until the START button is pressed (a programmed HOLD should not be confused with the HOLD button).	**Edit Program:** **Add Phase: HOLD**

Phase: PURGE

Description

This function is designed to warm up the Gas delivery head as quickly as possible. It applies maximum power to the heaters in the Gas delivery head to get to 300 K as quickly as possible. The PURGE is replaced by a SOAKING as the heaters run for a further 10 minutes.

Edit Program:
Add Phase: PURGE

Phase: END

Description

System shutdown. You are asked to enter a Ramp Rate back to a final temperature of 300 K and then the system is shut down. This is the controlled way to finish an experiment and should be used whenever possible.

Once an END function has been programmed, it is not possible to enter any more phases. The only options available are to load or save a program or delete the last phase.

Edit Program:
Add Phase: End

Ramp Rate:
120K/hr

Delete Phase

Description

To delete a phase at any time, in Program Mode, spin the IntelliKnob to 'Delete Phase' and press ENTER. This will delete the last phase entered. If the system happens to be executing this last phase, the phase will be replaced by a HOLD.

Edit Program:
Delete Phase

Load Program/Save Program

Description

While the system is idle, it is possible to load or save a program. Loading a Program simply loads the last saved program.

Edit Program:
Load Program

Edit Program:
Save Program

NOTE

Turning the IntelliKnob also offers the option to load a program or save the current program. This is only possible when the system is idle.

Press ENTER on completing each screen. To cancel programming at any time, press PROGRAM or Display, (the PROGRAM lamp will go out).

Once the phases have been entered, press START to begin the first phase in the Phase Table. The controller will automatically enter a HOLD phase at the end of the program if one has not already

been programmed. If the system is running and already in a HOLD phase, pressing the HOLD button will pass to the next instruction in the Phase Table.

3.2.1 Ramping to high temperatures from very low temperatures

Due to the refrigeration power of the Cobra cooler, we recommend that if a user has been running the Cobra below 100 K but then wishes to Ramp the Cobra up to temperatures exceeding 150 K, they run the system at 100 K for 30 minutes, first. This will allow any excess refrigeration in the unit to be removed. This will avoid any re-condensation of the nitrogen gas.

3.2.2 Phase table

Press PROGRAM at any time during running to look at the Phase Table and enter more phases (see Table 3). This will enter *Display Mode 6* but will not give the option of loading or saving a program while the system is running.

If there is a list of phases longer than the screen in the Phase Table, this will be indicated by small characters on the left of the STATUS screen pointing up or down.

↑ ↓	This indicates it is possible to scroll up and down through the phases using the IntelliKnob.
↓	This indicates it is possible to only scroll down through the phases using the IntelliKnob.
↑ ⊥	This indicates that the end of the Phase Table has been reached and it is only possible to scroll up through the phases using the IntelliKnob.
⊤	This indicates that the top of the Phase Table has been reached.
>	This indicates the current programme running.

Table 3 – Phase table

3.2.3 HOLD and 'Un-HOLD'

A program can be paused at any time using the HOLD button, this will illuminate the HOLD lamp. To continue the program simply press the HOLD button at any time and the HOLD lamp will go out.

It is also possible to release the HOLD phase by pressing the START button.

3.2.4 Alarm conditions

The 700 Series style controller has a number of safety features. If there is an issue with the system, an alarm condition is indicated by an illuminated ALARM lamp and a warning will appear on the bottom screen of the controller. If any of these alarms arise, please refer to the Troubleshooting Guide in Section 9 or contact technical support at Oxford Cryosystems (support@oxcryo.com). The following list describes all the warnings stored by the controller.

Temp Warning

If the temperature error has reached 10K the controller will indicate a warning but will not shut down.

Pressure Warning

If a blockage forms inside the gas delivery head, the controller will sense the back pressure and trigger a pressure warning. To remove this ice blockage, restart the system and program a PURGE. A PURGE will heat up the system as quickly as possible.

Self-Check Fail

During the initialisation, the controller checks a variety of parameters to make sure that everything is connected properly and that there is continuity in all parts of the system. Try restarting the controller a number of times to see if the problem persists. If it does, contact Oxford Cryosystems (support@oxcryo.com).

Flow Rate Fail

As the controller is controlling the flow of gas through the system it will indicate if there is a gas flow problem. This could be due to a blockage or restriction, no source gas or an outward leak of source gas.

Temp Control Err

If the temperature error has reached 25 K and the controller reads this value five times from the system, the controller will indicate a warning and shut down.

Temp Reading Err

The controller received a nonsense reading from the temperature sensors.

Sensor Fail

If the controller received extreme values from the sensors, it will try to reset them. If the sensors fail to reset after five attempts, the controller will shut down with this error.

Brown Out

If there is a brief interruption in the electrical supply to the controller, the controller will indicate a 'Brown Out' has occurred. The controller will continue to function normally.

Sink Overheat

If the controller overheats, there will be a Sink Overheat warning. This is often due to the covering of the fan on the underside of the controller.

PSU Overheat

If the controller overheats, there could be a PSU Overheat warning. This is often due to the covering of the fan on the underside of the controller.

Power Loss

When the power to a controller is cut, the controller will report a Power Loss error in the diagnostic screen when it is restarted.

Refr Too Cold

The Cobra refrigerator has a lower temperature limit to prevent the nitrogen from freezing. If the temperature drops below that set point, the refrigerator is too cold.

Refr Time Out

The Cobra refrigerator is designed to reach its base temperature within a known period of time. If, for any reason, the refrigerator fails to get cold, it will time out and shut down. A likely cause of a time out is a poor vacuum.

Cryodrive Off

The controller is connected to the refrigerator compressor (Cryodrive). If this connection is broken, the Cryodrive is switched off or there is no power to the Cryodrive, the controller will indicate it.

Cryodrive Error

There are a number of warnings given out by the Cryodrive. These include temperature warnings and pressure warnings. A temperature warning may arise due to the water temperature being too hot or too cold. A pressure warning may arise from a low helium pressure.

Vac Gauge Fail

If the vacuum gauge on a Cobra becomes disconnected or if there is a fault with the gauge, the controller will indicate a problem.

Poor Vacuum

When the Cobra controller is first switched on and initialises it checks the reading from the vacuum gauge. The poor vacuum warning threshold is set to $10x10^0$ (10) mbar. **This is only a warning and will not prevent the Cobra being used.** However, under these circumstances it may take longer to cool the refrigerator which could result in a time-out error on the cooling of the unit. To avoid this, program a RAMP to 295 K at 120 K/hr and wait for the RefrT (refrigerator temperature) to drop below 80 K. Then, program a RAMP to the desired temperature at the desired rate. Make sure the system is running at 5 L/min and not 10 L/min.

See Section 7.2 - Pumping down the vacuum space in the Cobra.

NOTE

When there is a warning or if the system shuts down, the ALARM lamp will flash quickly and the buzzer will sound. See the diagnostics by pressing DISPLAY and report these to Oxford Cryosystems.

3.2.5 Safety features during power failures

The Cobra controller is designed to protect itself and the sample during power interruptions.

It is possible for the controller to maintain gas flow and not reset the controller during electrical interruption of between 0-2 seconds. If a 'Brown-Out' is detected, this is indicated on the screen.

3.2.6 Checklist to start the Cobra running

Double check:

- ✓ The controller is switched on.
- ✓ Cryodrive has adequate water-cooling and switched on
- ✓ Nitrogen gas is connected to the back of the controller, set to 1 bar pressure and there is adequate supply

3.3 How to shut down the Cobra

It is worth noting here that the Cobra system performs best under continuous operation. The vacuum system in the Cobra is maintained more strongly when the unit is Cryopumping itself, i.e. when the heat exchanger is cold.

To shut down the Cobra correctly, the user should program an END phase.

Once the Cobra has shut down it is necessary to press the START button in the front of the controller if one wishes to continue using the system.

In the case of an unexpected shut down, press the START button, wait for the system to initialise and the record all the information on the status screen before switching the Cobra controller off.

Once the controller is shut down, the Cryodrive can be switched off at the main switch on the front panel and, if necessary, the water can be turned off.

NOTE

The Cryodrive will come on by itself if it is not plugged into the Cobra controller. The Controller controls the operation of the Cryodrive and when it is plugged into the controller, the Cryodrive is held in the 'off' state.

3.3.1 Cobra shut downs

The Cobra Controller has been designed to fully protect the Cobra under its normal mode of operation as outlined in this manual. The control program will shut down the Cobra if:

- The Gas Temp thermometer registers an error greater than ±25 K.
- The controller overheats.
- A temperature sensor (Gas temperature or Shield temperature) fault occurs.
- A Cobra refrigerator cable fault is detected.
- The Cryodrive shuts down unexpectedly (low charge pressure/lack of water cooling resulting in high temperature)

In each case the Status Screen variables are fixed and a suitable error message is displayed.

4 How to get the best performance from Cobra

To ensure that the Cobra performs fully, leaving the vacuum running between periods of use will help maintain the vacuum, but will also increase the Cobra's running hours, bringing the system closer to its service interval.

Repeatedly switching the system on and off may result in a need to re-pump the vacuum more regularly. You may expect the vacuum to last 5-6 months between pump downs when switching the system off less frequently. However, if the system is switched on and off repeatedly, it may be necessary to re-pump the system every few months.

5 Running the Cobra with Cryopad

Cryopad is a PC program which allows remote monitoring and control of any 700 series Oxford Cryosystems device. This includes the 700 series Cryostream / Plus, Cobra / Plus, PheniX and N-HeliX systems.

5.1 Installing Cryopad

Install Cryopad from the CD also supplied with the Cryostream or download from the Oxford Cryosystems website, www.oxcryo.com/software/cryopad. If you experience problems with the web installer please install the Microsoft Visual C++ Redistributable Package as explained on the above web site.

5.2 Using Cryopad to run the Cobra

With the controller switched off connect a COM port from your PC to the port labelled SERIAL on the back of the controller. **This connection can be made with any standard M-F serial cable**. Now turn on the contoller and start Cryopad by selecting from the Oxford Cryosystems group in the Start Menu, or else by double-clicking the Cryopad logo on your desktop.

5.2.1 Connecting using the Settings page

The first time you use Cryopad you will need to select a COM port using the Settings page. If you know which COM port you are using then select it from the **Connect Using Port** menu. If you are using a non-standard COM port you may type its name directly here. Alternatively click the **Search…** button to display the Search dialog, which may be used at any time to scan your computer's COM ports for compatible devices.

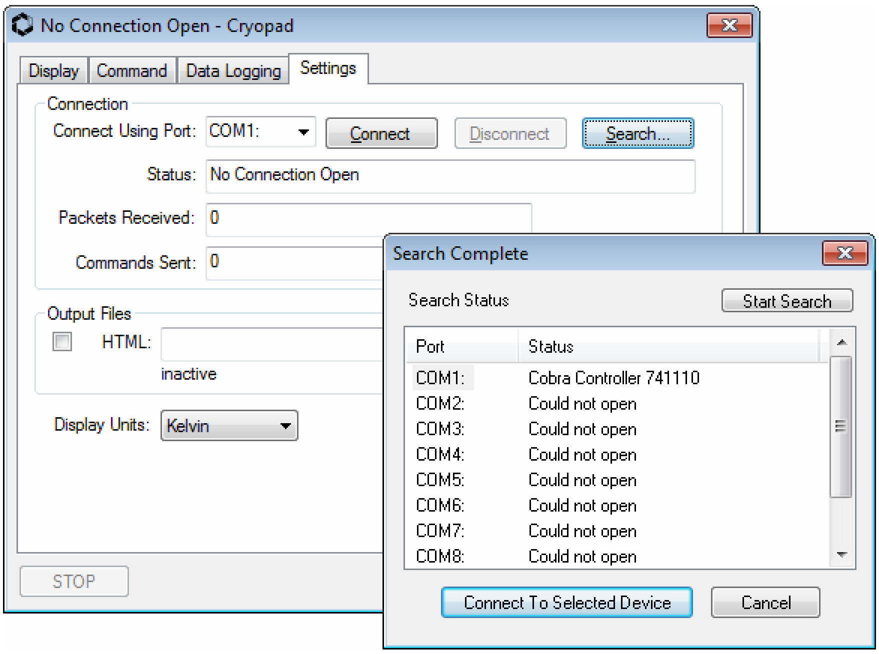


Figure 12 – Cryopad Settings page and Search dialog

The next time you run Cryopad your chosen COM port will be opened automatically, and a connection will be established as soon as a compatible device is detected. Should you need to change the COM port, switch to the Settings page, press Disconnect and repeat the above procedure. Should you wish to connect more than one device, run a new copy of Cryopad for each device and connect each one as described above.

5.2.2 The Display page

Once a connection has been established the Display page shows the live status of the device. The table below indicates the meaning of the various quantities displayed. After half an hour or so when the device has reached its normal operating values all the indicators will appear green.

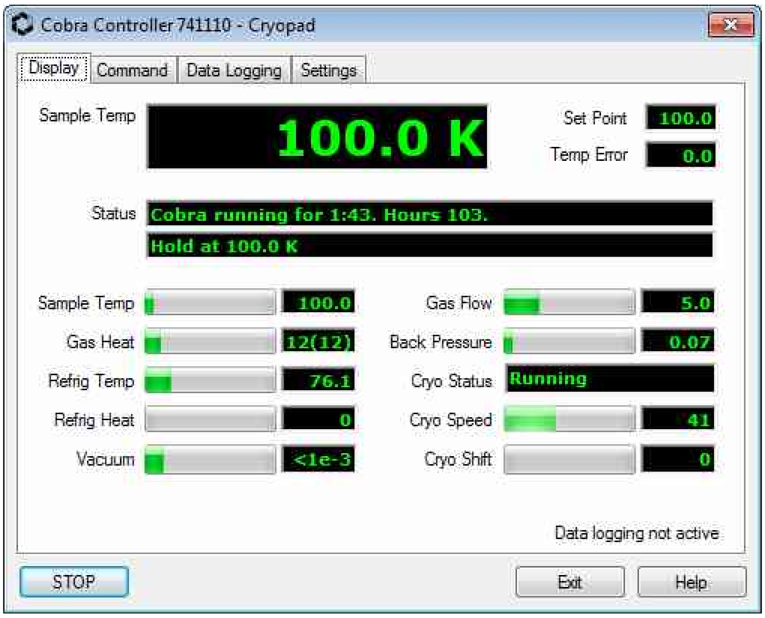


Figure 13 – Cryopad Display page

Data	Explanation
Sample Temp	The gas temperature at the crystal position.
Set Point	The temperature to which Cobra is controlling.
Temp Error	The difference between the Sample Temp and the Set Temp, except in a Cool phase, in which Temp Error is zero.
Status	Two lines of information indicating the current status of Cobra. Any errors or warnings raised by Cobra will be displayed here.
Gas Heat	The power to the sample heater, expressed as a percentage of full power.
Refrig Temp	The temperature measured at the refrigerator.
Refrig Heat	The power to the refrigerator heater, expressed as a percentage of full power.
Vacuum	The pressure in mbar measured by the vacuum gauge.
Gas Flow	The gas flow in litres / minute. The letter T indicates that Cobra is in Turbo mode.
Back Pressure	The back pressure in the gas line, measured in bar.
Cryo Status	An indication of the status of the Cryodrive
Cryo Speed	The current Cryodrive speed, presented in RPM.
Cryo Shift	A parameter indicating the amount by which the Cryodrive speed has been increased to allow very low temperatures to be attained.

5.2.3 The Command page

The Command page allows commands to be sent to your Cobra exactly as if they were entered using the controller. Commands sent in this way will immediately overwrite the contents of the controller's Phase table. Refer to section 3 above for details of the commands.

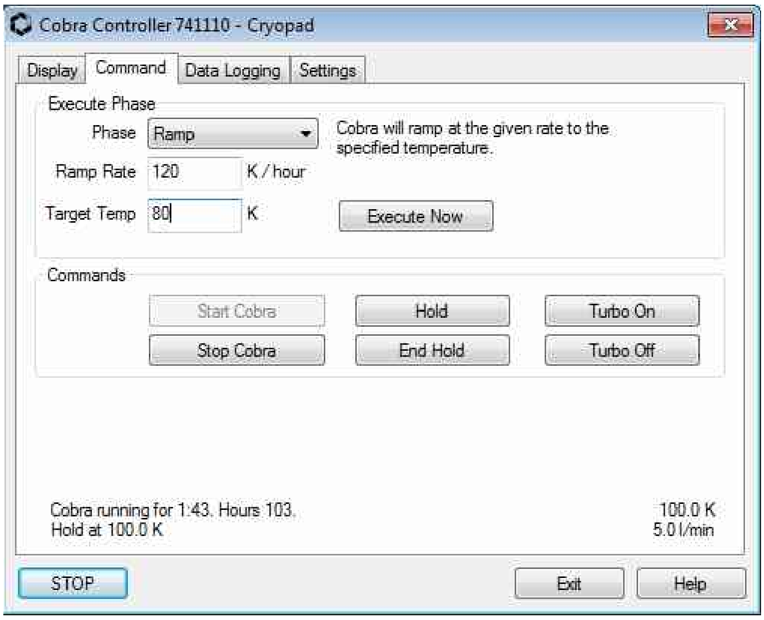


Figure 14 – Cryopad Command page

5.2.4 The Data Logging page

Cryopad allows data to be logged to a tab-delimited text file suitable for use in Excel or similar programs. Choose a file to which the data will be logged using the Log File item, and select the quantities of interest using the check boxes. The Interval item allows you to select the interval in seconds at which the data are logged. For monitoring purposes a 60 s interval is suitable whereas for diagnosing problems an interval of 1 s provides the most information but will produce a larger log file. Check the Logging Active box to commence logging.

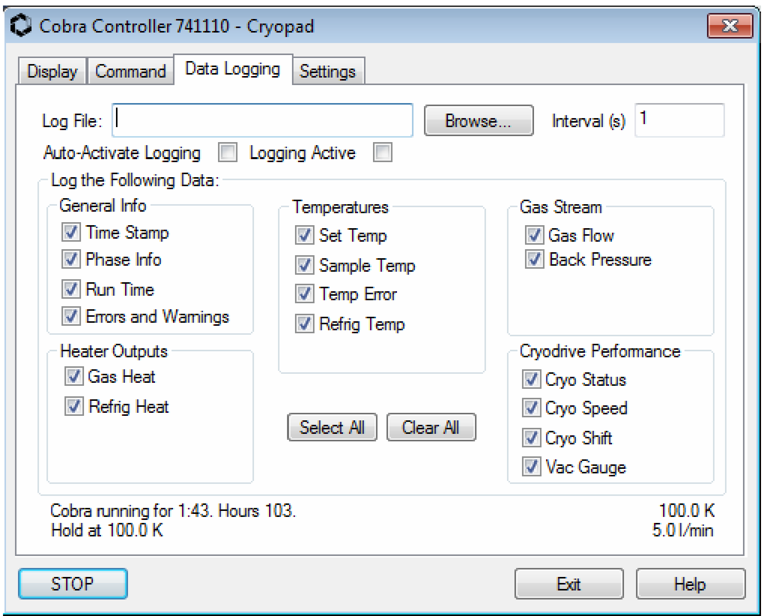


Figure 15 – Cryopad Data Logging page

6 Measuring the true crystal temperature

NOTE

We do not recommend measuring the temperature with a thermocouple placed in the stream.

In the heterogeneous environment of a narrow cold gas stream, there are several factors that lead to spurious voltages on the thermocouple, creating errors in apparent temperature of possibly tens of degrees! For instance, conduction of heat down the wires creates a heat leak. At the point of entry of the thermocouple wires into the stream a cold junction is formed whose temperature is much lower than the room temperature assumed by the controller, thus making the measured temperature *appear* to be much higher than indicated. Also, the sharp temperature change at the interface between the cold stream and the surrounding warm air can induce stresses into the thermocouple wires and then generate spurious EMFs.

We believe that the only satisfactory way to find the error in absolute temperature at the crystal position is to calibrate with a sample that undergoes a known phase transition or change of state. For instance, we have found that the low-temperature phase transition in the langbeinite $(NH_4)_2Cd_2(SO_4)_3$ was observed from intensity measurements to be in the range 88-89 K (established elsewhere to be at 88 K). Similarly, lattice parameter measurements of sodium ammonium tartrate tetrahydrate gave a transition temperature in agreement to within 0.5 K of the published value of 109 K. A most useful compilation of transitions in hundreds of crystals has been published by P. Tomaszewski (*Phase Transitions*, **38**, 127).

7 Cobra user maintenance

7.1 Preventative maintenance

The Cobra has been designed to be as easy to use as possible and should run without the need for constant attention. However, it may be necessary for the user to perform certain preventative service tasks to ensure the unit performs properly. These may include the following:

Procedure	**Service Interval**
Pumpdown of vacuum inside the Cobra refrigerator	Annually
Replacement of Line Drier	12,000 Hours or as required
Top up the helium pressure in the Cryodrive gas circuit	As required, see Section 7.7
Parker nitrogen generator inlet filter replacement	8,000 Hours
Parker nitrogen generator re-calibration of the oxygen sensor	Annually or as required

Table 4 – Cobra Service Intervals (minor service, user serviceable)

Approximately every 10,000 to 12,000 hours, a full service should be performed on the Cobra. The Oxford Cryosystems technical support team can arrange for an on-site visit by one of its engineers or in some cases a local agent may be able to perform the work in conjunction with Oxford Cryosystems. This service would normally involve the following:

Procedure	**Service Interval**
Cobra refrigerator Coldhead swap-out	12,000 Hours
Replacement of Cryodrive Adsorber	12,000 Hours
Parker nitrogen generator ; replacement of oxygen sensor	Every 3 years
Parker nitrogen generator; replacement of compressors (2x)	24,000 Hours or 3 years

Table 5 – Cobra Service Intervals (major service, training required)

Please note that the service intervals given above are typical of a standard Cobra usage. However, Cobra usage varies from customer to customer; from 1000 to 8000 hours usage per year. Therefore please contact Oxford Cryosystems to discuss how these intervals may fit in with your standard maintenance routines.

Service contract packages are also available for the Cobra system and more information is available upon request by contacting us at support@oxcryo.com.

Before starting any work you must do the following:

- Ensure only a suitably trained and supervised technician performs maintenance work.
- Wear the appropriate safety clothing.
- Check that all the required parts are available and are of the correct type before starting work.
- Isolate the Cobra and Cryodrive from the electrical supply so that it cannot be operated accidentally; if possible, remove the fuse from your electrical supply current and lock the isolator switches to the OFF position.

WARNING

Obey the safety instructions given above and below and take note of appropriate precautions. If you do not, you can cause injury to persons and damage to equipment.

7.2 Pumping down the vacuum space in the Cobra

You will find that from time to time you may need to re-pump the vacuum insulation space of the Cobra refrigerator. This will be apparent when the outside of the gas delivery head, refrigerator and transfer line becomes excessively cold or wet all over during operation or the Cobra is unable to reach the required gas temperature and the Heat% value is zero.

Do not be misled by similar symptoms that are not due to a vacuum problem.

NOTE

Leaving the vacuum running between periods of use will help maintain the vacuum, but will also increase the Cobra's running hours, bringing the system closer to its service interval.

It has been established that the Cobra vacuum can be re-pumped using a good rotary vacuum pump (with air ballast valve) and a Pirani gauge. It is not necessary to use a diffusion pump or turbomolecular pump – in fact, backstreaming of diffusion pump fluid could contaminate the system.

The correct procedure is as follows:

1. Determine that the unit really does require re-pumping as described above (see Troubleshooting Guide in Section 9 or contact Oxford Cryosystems or your local agent if you are unsure.)
2. Switch the Cobra off and leave it to warm up. Allow plenty of time for the unit to warm up internally (overnight), or run a PURGE program.
3. Screw the pump-out adaptor onto the pump-out port (see Figure 16). Connect a good rotary pump (preferably 2-stage) to the Cobra pump-out port using the pumping adaptor (see picture below) supplied.

   Do not withdraw the sealing plug at this stage.

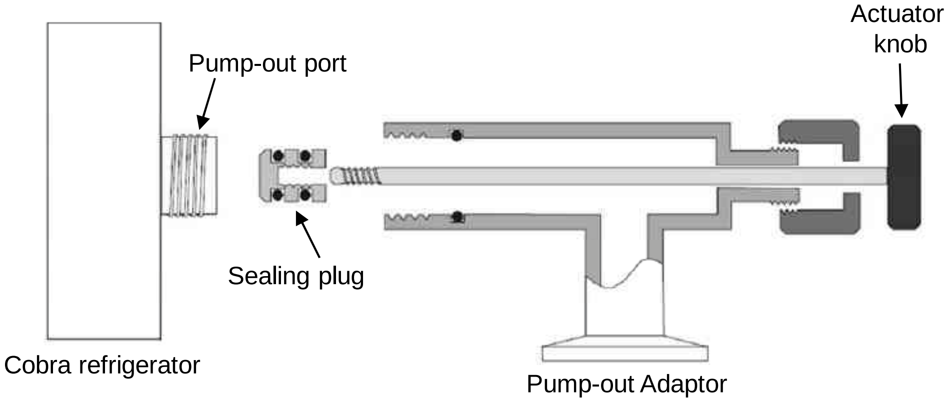


Figure 16 – Attaching the pumping adaptor

4. Start the rotary pump and ensure that a pressure of 0.1 mbar can be obtained up to the pumping adaptor. It may be necessary to run the pump with its air ballast valve open for about 30 minutes.

5. When the rotary pump pressure is 0.1 mbar, screw the pumping adaptor actuator knob into the sealing plug. NOTE: Do not screw the actuator knob into the sealing plug tightly as it will be more difficult to unscrew when being removed. Withdraw the sealing plug very slowly to avoid a rush of gas by pulling the actuator knob out. To secure the actuator knob in place, tighten the black knurled nut on the pump-out adaptor.
6. Wait until the pressure falls towards 0.1 mbar (this may take 60 minutes). If necessary, use the rotary pump air ballast again. If you cannot obtain a sufficiently low pressure at this stage you may have a leak (or bad pump!) which should be investigated. Contact your Cobra supplier if all else fails.
7. The vacuum should be pumped for an absolute minimum of 16 hours, and preferably for up to 72 hours where possible. The final pressure should be 0.1 mbar (or better) with a 2-stage rotary pump. You may well achieve 0.01 mbar but do not worry too much as Pirani gauges are not always very accurate. Switch on the Cobra controller and press DISPLAY. Turn the knob to vacuum to get an additional real-time reading of the vacuum inside the Cobra.
8. To finish pumping, the sealing plug should be pushed back in using the actuator knob of the pumping adaptor.
9. Unscrew the actuator knob from the sealing plug and remove the pumping port adaptor. Replace the pumping port cover.

7.3 Coldhead swap-out

The coldhead located inside the Cobra refrigerator will eventually require a service due to the delicate seals and ‘O’ rings wearing out. The easiest and most efficient way to do this is to contact Oxford Cryosystems for a replacement coldhead swap-out.

7.4 Changing inlet filters in the nitrogen generator

The nitrogen generator supplied by Oxford Cryosystems is a Parker NitroFlow Lab Cryo system (if purchased along with a Cobra). The NitroFlow Lab is set up to notify the user to change the air inlet filter every 8,000 hours. Please contact Oxford Cryosystems (support@oxcryo.com) for spare parts and a Tech Note describing how to re-set this alarm.

7.5 Calibrating or replacing the oxygen sensor in the nitrogen generator

From time to time the Oxygen Sensor located inside the Parker NitroFlow Lab nitrogen generator will need to be calibrated by the user. For instructions please refer to the Parker Manual supplied with the generator. This should be carried out once a year and the sensor will need to be replaced at least every 3 years. For further information and spare parts please contact us at Oxford Cryosystems.

7.6 Replacing the Cryodrive Adsorber

When the Cryodrive has been operating for approximately 12,000 hours, you must replace the Adsorber with a new one to avoid permanent damage to the Cobra refrigerator. Please contact Oxford Cryosystems for more information.

The new Adsorber is supplied pressurised with helium, so you should not have to re-charge the Cryodrive with helium after you fit the new Adsorber. A de-pressurisation adapter is supplied with the new Adsorber.

If necessary, refer to the ‘Cryodrive Operation & Instruction Guide’ for details of how to connect and disconnect the self-sealing Aeroquip coupling used for the Adsorber in the Cryodrive.

WARNING

Do not bend over the internal pipe work when you fit and remove the Adsorber. After removal, the old Adsorber must be safely depressurised before disposal. The replacement Adsorber will be charged with helium to 16.5 bar. Always vent gas safely, directed away from personnel.

Replacing the Adsorber:

1. Switch the Cryodrive ON/OFF switch to OFF, isolate the Cryodrive from the electrical supply.
2. Remove the lid of the Cryodrive.
3. Disconnect the helium supply hose located at the rear of the Cryodrive.
4. Disconnect the Aeroquip coupling from the Adsorber inlet.
5. Unscrew and remove the Adsorber rear panel locking nut and washer. Remove the single screw retaining the Adsorber inlet connection clamp plate. Lift the Adsorber out of its locating hole and remove the Adsorber. Remove the locking nut and clamp plate from the Adsorber inlet connection. Retain the clamp plate and screw, locking nut, washer and star washer.
6. Depressurise the old Adsorber by connecting the depressurisation adaptor to the Adsorber helium inlet and outlet coupling and tighten slowly by hand.
7. Remove the dust from the inlet and discharge self-sealing couplings of the new Adsorber. Fit the Adsorber clamp plate and locking nut to the Adsorber Aeroquip inlet connection.
8. Install the new Adsorber in position in the compressor unit and ensure that the locating pin is correctly engaged. Secure the new Adsorber in place using the nut and washers, and Adsorber clamp plate screw retained in step 5.
9. Re-connect the helium supply hose. Re-connect the internal Aeroquip on the Adsorber inlet.
10. Re-fit the lid of the Cryodrive.

Check that the pressure gauge reads 16.5±1.0 bar (239.31±14.5 psig). If the gauge reads below 15.5 bar, add helium gas following the procedure detailed in Section 7.7.

►**Record the Cryodrive running hours and date that the new Adsorber is fitted.**

7.7 Topping up the Cryodrive with helium

The Cobra refrigerator system consists of a closed 16.5 bar helium gas circuit. The helium in this circuit will eventually leak out over time and will require topping up from a high pressure helium gas cylinder (99.9995% purity). Each Cobra is supplied with a Cryodrive top up valve, which should be used to connect the cylinder to the front of the Cryodrive, via a single-stage high pressure helium regulator and a suitable hose. Ensuring that the tubing to the fitting is purged with helium first, the cylinder can be opened and the helium can be quickly topped up to 16.5 bar. **Note that this should only be performed when the system is off.**

Re-charge the Cryodrive with helium if the helium pressure falls to below 15.5 bar. If you need to re-charge the Cryodrive frequently (for example, every 6 months or more often), there is probably a leak in your installation. Use a helium leak detector or other suitable method to find the leaks, and contact Oxford Cryosystems with the results, or for further guidance.

CAUTION

Ensure that the interconnecting pipe work is capable of safely withstanding the maximum regulator delivery pressure.

You must re-charge the Cryodrive with 99.9995% helium. If you do not, you will contaminate the Cryo with impurities that will reduce its efficiency.

To recharge the system with Helium, please follow the instructions in Section 5.3 of the Cryodrive manual, supplied with the Cryodrive.

Note that if the helium pressure has fallen to 0 bar (atmospheric pressure), the helium should not be topped up. Instead, contact Oxford Cryosystems for advice on how to proceed.

8 Liquid and gaseous nitrogen safety sheet

8.1 General

These safety points are a guideline to outline the potential hazards and procedures involved in the handling of liquid or gaseous nitrogen. Anyone handling liquid or gaseous nitrogen should first inform their departmental or laboratory safety advisor and receive advice about local safety procedures.

All users are requested to read this safety sheet before handling liquid nitrogen. Oxford Cryosystems accept no responsibility for injury or damage caused by the mishandling of liquid or gaseous nitrogen.

8.1.1 General properties

- Gaseous nitrogen is colourless, odourless and tasteless and is slightly lighter than air at equal temperatures; cold nitrogen vapour is, however, denser than atmospheric air.
- Liquid nitrogen is odourless, colourless and boils at -195.8°C. One volume of liquid nitrogen gives approximately 700 volumes of gas at ambient conditions.
- Nitrogen is not flammable. It is chemically inert, except at high temperatures and pressures. Its volume concentration in air is 78%.
- Liquid and cold gaseous nitrogen can cause severe burns or frostbite when in contact with the skin or respiratory tract.
- Gaseous and liquid nitrogen is non-corrosive.
- Nitrogen does not support life and acts as an asphyxiant.
- Nitrogen is intrinsically non-toxic.

8.2 Fire and explosion hazards

Gaseous and liquid nitrogen are non-flammable and do not, themselves, constitute a fire or explosion risk. However, both gaseous and liquid nitrogen are normally stored under pressure and the storage vessels, whether gas cylinders or liquid tanks, should not be located in areas where there is a high risk of fire or where they may normally be exposed to excessive heat.

8.3 Health hazards

8.3.1 Asphyxia

Nitrogen, although non-toxic, can constitute an asphyxiation hazard through the displacement of the oxygen in the atmosphere. Nitrogen gas or oxygen depletion is not detectable by the normal human senses.

Oxygen is necessary to support life and its volume concentration is the atmosphere is 21%. At normal atmospheric pressure persons may be exposed to oxygen concentrations of 18% or even less, without adverse effects. However, the response of individuals to oxygen deprivation varies appreciably. The minimum oxygen content of breathing atmospheres should be 18% by volume but to ensure a wider margin of operational safety it is recommended that persons are not exposed to atmospheres in which the oxygen concentration is, or may become, less than 20% by volume.

Symptoms of oxygen deprivation, such as increased pulse and rate of breathing, fatigue, and abnormal perceptions or responses, may be apparent at an oxygen concentration of 16%.

Permanent brain damage or death may arise from breathing atmospheres containing less than 10% oxygen. Initial symptoms will include nausea, vomiting and gasping respiration. Persons exposed to such atmospheres may be unable to help themselves or warn others of their predicament. The symptoms are an inadequate warning of the hazard.

WARNING

Breathing a pure nitrogen atmosphere will produce immediate loss of consciousness and almost immediate death.

8.3.2 Cold burns

Liquid and cold nitrogen vapours or gases can produce effects on the skin similar to a burn. Naked parts of the body coming into contact with un-insulated parts of equipment may also stick fast (as all available moisture is frozen) and the flesh may be torn on removal.

8.3.3 Frostbite

Severe or prolonged exposure to cold nitrogen vapour or gases can cause frostbite. Local pain usually gives warning of freezing but sometimes no pain is experienced. Frozen tissues are painless and appear waxy with a pallid yellowish colour. Thawing of the frozen tissues can cause intensive pain. Shock may also occur if the burns are at all extensive.

8.3.4 Effect of cold on Lungs

Prolonged breathing of extremely cold atmospheres may damage the lungs.

8.3.5 Hypothermia

Low environmental temperatures can cause hypothermia and all persons at risk should wear warm clothing. Hypothermia is possible in any environmental temperature below 10ºC but susceptibility depends on time, temperature and the individual. Older persons are more likely to be affected. Individuals suffering from hypothermia may find that their physical and mental reactions are adversely affected.

8.4 Precautions

8.4.1 Operations and maintenance

It is essential that operations involving the use of gaseous or liquid nitrogen particularly where large quantities are used are conducted in well-ventilated areas to prevent the formation of oxygen deficient atmospheres.

Ideally, nitrogen should be vented into the open air well away from areas frequented by personnel. It should never be released or vented into enclosed areas or buildings where the ventilation is inadequate. Cold nitrogen vapours are denser than air and can accumulate in low lying areas such as pits and trenches.

Where large spills of liquid nitrogen occur a fog forms in the vicinity of the spill caused by the condensation of water vapour in the surrounding air. The fog, in addition to severely reducing visibility, may contain oxygen concentrations appreciably lower than that of the air presenting a local asphyxiation hazard.

8.4.2 Personnel protection

Persons handling equipment in service with liquid nitrogen should wear protective face shields, loose fitting gauntlets and safety footwear.

8.4.3 Emergencies

In the event of an accident or emergency the instructions below should be implemented without delay.

8.4.4 Asphyxiation

Persons showing symptoms of oxygen deprivation should be moved immediately to a normal atmosphere. Persons who are unconscious or not breathing must receive immediate first aid. Medical assistance should be summoned without delay. First aid measures included inspection of the victim's airway for obstruction, artificial respiration and simultaneous administration of oxygen. **These procedures should only be carried out by trained first aid staff.** The injured should be kept warm and resting.

It is important that the personnel carrying out rescue operations should minimise the risk to themselves.

8.4.5 Treatment of cold burns and frostbite

Cold burns should receive medical attention as quickly as possible. However, such injuries are not an everyday occurrence and doctors, hospital staff or works first aid personnel may not be aware of the basic methods of treatment. The following notes describe the first aid treatment and recommended advice for further treatment to be given by a medical practitioner or a hospital.

8.5 First aid

In severe cases summon medical attention immediately. Flush affected areas of skin with copious quantities of tepid water to reduce freezing of tissue. Loosen any clothing that may restrict blood circulation. Move the victim to a warm place but not to a hot environment and do not apply direct heat to the affected parts. Every effort should be made to protect frozen parts from infection and further injury. Dry, sterilised bulky dressings may be used but should not be applied so tightly that blood circulation is restricted.

8.5.1 Treatment by medical practitioner or hospital

1. Remove any clothing that may constrict the circulation to the frozen area. Remove patient to sick bay or hospital.

2. Immediately place the part of the body exposed to the cryogenic material in a water bath which has a temperature of not less than 40°C but no more than 45°C. **Never use dry heat or hot water.** Temperatures in excess of 45°C will superimpose a burn upon the frozen tissue.

3. If there has been a massive exposure to the super cooled material so that the general body temperature is depressed, the patient must be re-warmed gradually. Shock may occur during re-warming, especially if this is rapid.

4. Frozen tissues are painless and appear waxy with a pallid yellowish colour. They become painful, swollen and very prone to infection when thawed. Therefore, do not re-warm rapidly if the accident occurs in the field and the patient cannot be transported to hospital immediately. Thawing may take from 15-60 minutes and should be continued until the blue, pale colour of the skin turns to pink or red. Morphine, or some potent analgesic, is required to control the pain during thawing and should be administered under professional medical supervision.

5. If the frozen part of the body has thawed by the time medical attention has been obtained, do not re-warm. Under these circumstances cover the area with dry sterile dressings and a large bulky protective covering.

6. Administer a tetanus booster after hospitalisation.

8.5.2 Hypothermia

Persons suspected to be suffering from hypothermia should be wrapped in blankets and moved to a warm place. Slow restoration of temperature is necessary and forms of locally applied heat should not be used. Summon medical attention.

8.5.3 Liquid nitrogen spillage

If large spills of liquid nitrogen occur, large quantities of water should be used to increase the rate of liquid vaporisation.

9 Cobra troubleshooting guide

During the operation of the Cobra it will be necessary to routinely check many system parameters, such as vacuum within the Cobra refrigerator, gas flow, Cryodrive compressor pressure, Cryodrive water-cooling and quantity of helium remaining. Good system maintenance and monitoring will help to avoid unexpected problems.

For a detailed Cryodrive fault-finding schedule, see the 'Cryodrive Operation & Instruction Guide' (supplied with your Cryodrive and available from Oxford Cryosystems)

9.1 Very important guidelines for using this document

1. This guide is designed for operators responsible for looking after the Cobra.
2. This guide is not designed to cover every technical eventuality but to provide the correct interpretation of, and solution to, a variety of common symptoms. As a user, symptoms may arise that are not covered here. If, at any time, you are unsure of the cause of the Cobra problem, contact your local agent or Oxford Cryosystems directly.
3. If you experience a shutdown or unusual behaviour from your system, please record as much information as possible and any physical symptoms you feel are a concern. Then contact Oxford Cryosystems or your local agent.
4. **Do not** rush into changing components or fixing something until you have spoken to Oxford Cryosystems (remember technical support and advice are free of charge) or your local agent. Changing components can **very often** create more problems and mask the original fault.

9.2 Problems and solutions

9.2.1 Condensation and/or ice covering the outside of the gas delivery head, the flexible transfer line or refrigerator

Cause

Condensation and/or ice over the outside of the Cobra transfer line or can indicate a loss of vacuum. This should not be confused with localised spots of ice or condensation. Remember, it's impossible to lose a vacuum from one small area of vacuum space! This loss of vacuum can be for two reasons:

1. Natural out-gassing over a period of time depending on the physical treatment of the gas delivery head.
2. Vacuum leak at an internal or external joint or cracked flexible line, although this is rare.

To confirm the quality of the vacuum as the source of the problem, press DISPLAY and turn the knob to display the 'Vacuum' reading. When running, the system should be in the 10^{-3} range.

Solution

Re-pump the vacuum, especially if this has not been done for over 12 months.

If it is believed there is a leaky joint or cracked flexible hose, contact Oxford Cryosystems or your local agent as this is not user serviceable.

Associated symptoms

Inability to reach low temperatures and the base temperature begins to rise (even giving a temperature warning) when using a COOL function

Cobra indicates 'Poor Vacuum' and ALARM lamp flashes

9.2.2 Cobra indicates 'Poor Vacuum' and ALARM lamp flashes

Cause

This is caused by a degrading vacuum insulation in the coldhead and is determined by the controller diagnostics. The poor vacuum warning is set off at 10 mbar ($10x10^0$) but should not prevent you using the system. Possible cause of this problem:

1. Natural out-gassing over time depending on the usage of the system.
2. Vacuum leak at an internal or external joint or cracked flexible line, although this is rare.

Solution

This is only a warning and will not prevent the Cobra being used. However, under these circumstances it may take longer to cool the refrigerator which could result in a time-out error on the cooling of the unit. To avoid this, program a RAMP to 295K at 120K/hr and wait for the RefrT (refrigerator temperature) to drop below 80K. Then, program a RAMP to the desired temperature at the desired rate. Make sure the system is running at 5l/min and not in TURBO mode (10l/min.)

Consult the section 7.2 on re-pumping the vacuum.

If it is believed there is a leaky joint or cracked flexible hose, contact Oxford Cryosystems or your local agent as this is not user serviceable.

Associated symptoms

- Inability to reach low temperatures and the base temperature begins to rise

9.2.3 Localised ice spot on the flexible line

Cause

This is due to the flexible line being bent too sharply beyond its 200 mm minimum radius so that the transfer capillary inside the flexible vacuum jacket touches the wall of the vacuum jacket.

Solution

The Cobra will continue to run happily like this, but it may be worth contacting Oxford Cryosystems or your local agent. Try to increase the bend radius at the point where the ice spot occurs.

9.2.4 Inability to reach low temperatures

Cause

If the gas temperature will not drop below a certain temperature (for example, the programmed gas temperature is 100 K and the Cobra will only reach 105 K but is stable and not rising,) or has been rising over a period of time, the vacuum may be poor or the system may require servicing.

Solution

If the base gas temperature that the Cobra reaches continues to rise, check to make sure the performance of the refrigerator system is adequate. This can be done by pressing DISPLAY and turn the knob to show the RefrT value. If this number is above 80 K then the refrigerator is not functioning properly. Pull down the cover over the front of the Cryodrive helium compressor to ensure that the gas pressure is correct. When the system is off and has cooled down, the helium gas pressure should be 16.5 bar.

If the performance of the refrigerator is adequate, it is likely that the vacuum is degrading and will require re-pumping. Consult Section 7.2 for this procedure.

Associated symptoms

Condensation and/or ice covering the outside of the gas delivery head or the flexible transfer line

Inability to reach low temperatures and the base temperature begins to rise (even giving a temperature warning) when using a COOL function

Cobra indicates 'Poor Vacuum' and ALARM lamp flashes

9.2.5 Inability to reach low temperatures and the base temperature begins to rise (even giving a temperature warning) when using a COOL function

Cause

These symptoms arise from the resultant heat loads introduced to the system by a COOL function meaning that the system is unable to successfully maintain its target temperature.

Solution

The way to avoid this problem is to first ensure the system is either waiting to be started (because the user has stopped it) or in a HOLD. If the system is maintaining its COOL function, press PROGRAM and turn the knob to 'Delete phase' and press ENTER. The system should be in a hold at the temperature it is currently sitting at.

Wait for the refrigerator to slow to its minimum and for the flow to increase to 5 L/min. Press DISPLAY and turn the knob to RefrT. This will reach its minimum which is typically between 75 K and 78 K.

If the system has been switched off, program a RAMP to 295 K at 120 K/hr and wait for the RefrT (refrigerator temperature) to drop below 80 K ensuring the flow remains at 5 L/min rather than 10 L/min. Then, program a RAMP to the desired temperature at the desired rate. Make sure the system is running at 5 L/min and not in TURBO mode (10 L/min.)

Eventually, it may be necessary to re-pump the vacuum if the temperature can't be obtained. Consult Section 7.2 for this procedure.

Associated symptoms

Condensation and/or ice covering the outside of the gas delivery head or the flexible transfer line

Inability to reach low temperatures

Cobra indicates 'Poor Vacuum' and ALARM lamp flashes

9.2.6 Gas temperature is unstable and the Cryodrive is speeding up and slowing down

Cause

This is a possible indication of a failing vacuum. To confirm this, look at the Vacuum reading on the Display screen by pressing the DISPLAY button. If the vacuum reading is higher than $1x10^{-3}$ mbar then some residual permanent products exist in the vacuum space.

Solution

Switch the Cobra off by pressing STOP or by performing a PURGE and leave the system so the RefrT value reaches and sits at room temperature for a couple of hours. Restart the system and program a RAMP to 295 K at 120 K/hr and wait for the RefrT (refrigerator temperature) to drop below 80 K. Then, program a RAMP to the desired temperature at the desired rate. Make sure the system is running at 5 L/min and not in TURBO mode (10 L/min.)

Associated symptoms

Condensation and/or ice covering the outside of the gas delivery head or the flexible transfer line

Inability to reach low temperatures and the base temperature begins to rise (even giving a temperature warning) when using a COOL function

Cobra indicates 'Poor Vacuum' and ALARM lamp flashes

9.2.7 Cryodrive fails to initialise when switched on

Cause

Each of the three LED indicators on the front of the Cryodrive should all come on and go out when the power switch is turned on. Assuming the Cryodrive is plugged into the Cobra controller, the Cryodrive should remain off. If the LEDs do not come on then it is likely there is a power problem.

Solution

There are several checks that can be made to determine if the problem can be easily rectified:

- Check the integrity of the fuse in the plug (if it has one).
- Look to ensure that the Cryodrive Protection Switch (positioned at the bottom in the centre of the unit) is in the ON position. This is indicated by the black button being depressed and the red button popped out.
- Check the two slow blow fuses in the side of the electrical control box.

If this fails to resolve the problem, contact Oxford Cryosystems Ltd.

9.2.8 Cryodrive initialises but fails to start when requested by controller

Cause

The water supply to the Cryodrive may be too cold. The minimum permissible water supply temperature is 8°C. There is a thermocouple on the pipe work around the compressor and if it reads a temperature lower than 8°C, the unit will not start even though it initialises.

Solution

Adjust the controlling temperature of the chiller to ensure that the water is controlling at a higher temperature.

If the water is very close to the desired temperature, (6-7°C) then it may be possible to start the unit by shutting off the water supply for a minute or so, start the system again and, once the unit is running, open the water supply. **Do not run the Cryodrive if the water is less than 6°C.**

9.2.9 Ice formation on the sample

Cause

Ice formation on the sample can begin at the point of flash cooling the sample or it can build up over time to eventually cover the sample and thus ruin the diffraction image.

NOTE

Ice on the sample does not come from the nitrogen gas travelling down the nozzle. Nitrogen gas from the Cobra is very dry (circa 0.1 ppm of water vapour).

Ice on the thin film supporting the crystal in the loop can arise from a number of sources.

1. Insufficient cryo-protectant of the buffer solution.

    a. Too much mother liquor results in dilution of the cryo-protectant to the point where it is no longer adequate.
    b. A thick film around the crystal may result in a larger thermal mass that must then be cooled.
2. Rate of flash cooling is too slow.
3. The sample is too far away from the nozzle or not aligned in the centre of the cold stream. The cold stream and the dry air stream mix and draw in atmospheric moisture that is frozen out on the sample.
4. The loop is unclean. Any particles on the loop will propagate ice formation.
5. A wet dry air supply or a disturbance of the laminar flow system due to drafts in your laboratory or an oversized sample mount (i.e. capillary or pin is too thick).
6. It is important that the velocities of the two gases (shroud and main stream) are the same. If they are grossly unmatched, atmospheric moisture will encroach the streams and cause ice to build up. A true laminar flow will prevent attack from atmospheric moisture.

Solution
To increase the effectiveness of the cryoprotectant, increase its concentration.

Position the nozzle as close to the sample as possible without affecting the path of the x-rays or casting an image on to the detector. The ideal position is inside the first 6mm from the end of the nozzle and the centre 2 mm diameter of the coldstream. Be sure to clean the loop before use, as ice build up will only compound the problem.

Check the laboratory for drafts. The most likely cause of turbulence is an air conditioning unit, or a cooling fan from an x-ray generator or the rotating anode generator. Create a screen between the source of the draft and your cold stream. This will greatly reduce the turbulence. If you are unsure of the source of the draft, try the Flashlight Test (see section 9.2.15).

Try adjusting the flow of the outer dry gas stream. 12-13 litres per minute is normally fine, but when the air is more turbulent, try turning the outer stream flow rate up to a maximum of 15 litres per minute; this can often cure the problem (see Section 9.2.15).

If the icing persists and there is also a concentric build up of ice on the nozzle, the most likely cause is a wet dry air supply. If you have an Oxford Cryosystems Dry Air Unit, change the Compressor Filter Delivery Element. If the icing persists, contact Oxford Cryosystems or your local agent about a Dry Air Unit service.

Associated symptoms
Concentric formation of ice around the nozzle

9.2.10 Ice formation on outer edge of the nitrogen gas cold stream nozzle

Cause
The likely cause of ice on one side only of the nitrogen nozzle is a misaligned dry air shroud tube.

Solution
Look up the nozzle of the Cobra and check to make sure the outer dry air shroud is concentric with the inner nitrogen nozzle. A small misalignment may be corrected by pushing the inner nozzle to one side. The shroud tube is locked into its 26mm diameter mounting bush using a low strength retainer compound (e.g. Loctite 222e). To release the shroud tube, grasp it gently and push to one side to release the retainer bond. Movement of the shroud tube will be limited as it touches the outside of the

inner nitrogen nozzle - this prevents the shroud tube from kinking. Once the outer dry air shroud has been removed, refit the shroud tube using a little retainer compound, check that the tube is concentric and allow the retainer to set.

9.2.11 Concentric formation of ice around the nozzle

Cause

This is likely to be wet air from the dry air supply or a high flow rate from the dry air supply. The cold stream requires a dry air shroud of dewpoint -60°C. If the stream is wet, the moisture in the air will freeze onto the nozzle and sample.

Solution

Contact Oxford Cryosystems or your local agent who will supply a Dry Air Unit service kit.

Associated symptoms

Ice formation on the sample or ice formation on the outer edge of the nitrogen gas cold stream head.

9.2.12 Positive gas temperature error

Cause

If after months of use, the Cobra starts to lose its vacuum insulation, then it will struggle to maintain its base temperature and the gas temperature error will increase, positively.

Solution

Re-pump your vacuum. Consult Section 7.2 for this procedure.

Associated symptoms

Condensation and/or ice covering the outside of the gas delivery head or the flexible Transfer Line

Inability to reach low temperatures and the base temperature begins to rise (even giving a temperature warning) when using a COOL function

9.2.13 Flow rate will not rise to 10 L/min when the TURBO button is pressed

Symptoms

The system is running and when the TURBO button is pressed, the flow does not rise from 5 to 10 L/min. The flow may stop at 7 or 8 L/min.

Cause

One of the likely causes of this problem is that the unit in the controller regulating and monitoring the flow is not functioning correctly. To test for this, do the following:

1. Switch the controller on.
2. Program in a RAMP to whatever the current room temperature default reading is (about 290-295 K) and press START.
3. Once the system has been running for a minute or so, press the TURBO button several times to cycle the flow rate between 5 and 10 L/min.
4. Press STOP when you have finished.

If the flow does not rise to 10 L/min then there is likely to be a problem with the flow controller.

Solution

Contact Oxford Cryosystems for a replacement flow controller.

9.2.14 EPROM fail on initialisation

Symptoms

During the initialisation of the controller, there is an EPROM failure.

Cause

The likely cause of this problem is corruption of the EPROM software. If this is the case, this problem can be rectified with the assistance of Oxford Cryosystems.

Take the lid off the controller, being careful of the earth wire that is attached inside when doing so. Move the jumper (JP9) to the SET pins and then turn the controller on.

Once the controller has finished initialising, you will see a list of parameters. Write down all the parameters and their corresponding values and send them to Support@oxcryo.com.

Solution

Oxford Cryosystems will then contact you for a solution.

9.2.15 Flashlight test

To be sure the flow rate of the outer dry air stream is correct, it is often better to set the flow by eye rather that by trying the guess what the flow should be by looking at the numbers.

Turn all the lights off in the x-ray room and shine a flashlight up towards the nozzle of the Cobra in an attempt to highlight the plume created by the cold gas stream. As the gas stream leaves the nozzle it is really made up of two parts; the first 'invisible' 10 or 12 mm and the remaining plume of ice. The object of the exercise is to maximise the length of the 'invisible' section. This should only be done over the first 15 L/min of air from the dry air source. One should not be fooled into thinking that at 25 L/min there is no plume, and therefore no ice, because the ice will build rapidly around the end of the nozzle and blow the sample from its support.

10 Technical support

10.1 Introduction

To allow Oxford Cryosystems to offer fast and accurate technical support, please quote your Cobra Serial Number with all technical issues. It is worth keeping a record of this number in a convenient place:

COBRA SERIAL NUMBER
This Cobra serial number is _______

Before you return your equipment you must warn Oxford Cryosystems by contacting us.

<u>Oxford Cryosystems Ltd contact details:</u>

Email: support@oxcryo.com

Phone: +44 (0)1993 883488

Fax: +44 (0)1993 883988

10.2 Returns procedure

Use the following procedure to return ANY items for repair.

1. Contact Oxford Cryosystems and obtain an 'RMA' number for your equipment which must be written on each box that you return. Without this number we may reject packages. You will also be emailed a form which you must fill in and email or fax back to us prior to sending your package(s).

2. Remove all traces of dangerous substances and any accessories that will be returned to Oxford Cryosystems. Drain all fluids and lubricants from the equipment and its accessories.

3. Disconnect all accessories from the equipment. Safely dispose of the filter elements from any oil mist filters.

4. Seal up all of the equipment's inlets and outlets (including those where accessories were attached). You may seal the inlets and outlets with blanking flanges or heavy gauge PVC tape.

5. Seal contaminated equipment in a thick polythene bag. If you do not have a polythene bag large enough to contain the equipment, you can use a thick polythene sheet.

6. If the equipment is large, strap the equipment and its accessories to a wooden pallet. Preferably, the pallet should be no larger than 510 mm x 915 mm (20"x 35"); contact Oxford Cryosystems if you cannot meet this requirement.

7. If the equipment is too small to be strapped to a pallet, pack it in a suitable strong box.

8. If the equipment is contaminated, label the pallet (or box) in accordance with laws covering the transport of dangerous substances.

Oxford Cryosystems - Warranty Certificate

This warranty is subject to the Oxford Cryosystems Ltd's (OCL) Terms and Conditions of Sale.

OCL warrants to the Buyer that the goods sold for use hereunder will be free from defects in material and workmanship under normal use and operation for 12 months from the date of shipment from OCL's premises.

In order to obtain the benefits of the warranty the Buyer must first notify OCL of the defects. An OCL representative will verify the nature of the defect and if it is covered by this warranty, OCL will issue the Buyer with a RMA number and provide the Buyer with instructions on how to return the goods to OCL. The Buyer must return the goods according to instructions from OCL, complete with a written description of the claimed defect and RMA number. The goods should be packed safely, preferably in its original packaging prior to return.

The Buyer shall meet the cost of shipping the defective goods to OCL and OCL will pay any return costs to the Buyer

OCL's obligation under this warranty is limited to its option to repair or replace goods that are proven to be defective when used under normal operating conditions and within specification.. This warranty does not cover any changes made by the customer, depreciation of the goods or claims for compensation.

No warranty is given for damage resulting from misuse or fair wear and tear. In addition, this warranty does not cover any costs incurred in damage arising from the dismantling or reassembly of any of the goods, or for consequential losses of time or materials caused by Cryostream failure.

Registration

In order for us to be able to provide fast and effective service, you should register your system with us. Please send the serial number of the system (found engraved on the Coldhead) to support@oxcryo.com, together with your full contact details.

To make contact with Oxford Cryosystems you can telephone, fax, or email us at:

Oxford Cryosystems Ltd
3 Blenheim Office Park
Lower Road, Long Hanborough
Oxford OX29 8LN, UK
Tel: +44 1993 883488 Fax: +44 1993 883988
Email: info@oxcryo.com